SYNTHETIC AND STRUCTURAL STUDIES WITH SOME AMIDES

A SYNOPSIS

OF THE THESIS TO BE SUBMITTED

TO

THE FACULTY OF SCIENCE AND TECHNOLOGY OF

MANGALORE UNIVERSITY

FOR AWARD OF THE DEGREE OF

DOCTOR OF PHILOSOPHY

IN

CHEMISTRY

BY

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Title of the Thesis **: SYNTHETIC AND STRUCTURAL STUDIES WITH SOME AMIDES**

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The thesis describes the synthetic, spectral and structural studies on a number of N-(aryl)-succinamic acids and succinamides. It analyses the results to explore the effect of substituents on the structural parameters and on the pattern of hydrogen bonding in these compounds. It further describes the results of synthetic and crystal structural studies of some of the N-(aryl)-alkylsuccinamates and N-(aryl)-succinimides with the same objective.

Spectroscopy and its applications form a significant part of modern chemistry and physics[1]. Spectroscopic technique is one of the most powerful tools available for the study of atomic and molecular structures and is used in the investigation of a wide range of samples. Infrared spectroscopy provides useful information about the structure of a molecule quickly and convincingly without tiresome evaluation methods. The direct applications of NMR studies are to provide evidences relating to the nature of the chemical bonding in molecules or crystals.

A knowledge of structure of materials is essential for a proper understanding of their physical and chemical properties. Thus crystal structure studies[2] have been extensively used to investigate the structural aspects of a variety of compounds. When a beam of X-rays is incident on matter, the electrons in the atoms interact with X-rays and hence the diffraction pattern gives information about the time averaged electron density. The pattern of scattered X-rays produced by a `good quality' single crystal contains spots of varied intensity. The pattern of spots reflects properties of the crystal structure. The pattern has a particular 'geometry' which is related to the lattice and unit cell geometry of the crystal and hence gives information about the distance between atoms. The unit cell dimensions are related to the distance between molecules. The diffraction pattern is the Fourier Transform of the electron density. Thus the crystal structure studies have been extensively used to investigate the structural aspects of a variety of compounds.

Amides are one of the well known classes of organic compounds in which the hydroxy groups of carboxylic acids are replaced by amino groups. They are represented by the general structure R^1 –CO-NX-R^2, where X = H, OH, NR^3_2

COR^3, R^3 and R^1, R^2, R^3 = H, alkyl or aryl group. Amides are of fundamental chemical interest as conjugation between nitrogen lone pair electrons and the carbonyl π-bond results in distinctive physical and chemical properties. Addition of a third hetero atom either α or β to the amino nitrogen of the amide group as in hydroxamic acids, N-halo compounds and hydrazides, increases their chemical complexity, which has required the advent of modern physical techniques to unravel. Further, the amide moiety is an important constituent of many biologically significant compounds. Thus an understanding of formation, properties and reactions of amides is central to future development in such areas as polypeptide and protein chemistry. Many imides, hydroxamic acids and hydrazides exhibit pharmacological activity, which has further stimulated recent interest in their chemistry[3]. The chemistry of succinamic acids and succinamides are of interest as they show distinct physical, chemical and biological properties. Many aryl-succinamic acids and aryl-succinamides show pharmacological, fungicidal, herbicidal, antitumor and anti-inflammatory activities[4]. Hence the N-aryl succinamic acids and N-aryl succinamides are of interest in synthetic and biological chemistry.

As a part of efforts of correlating spectroscopic parameters with the chemical bond parameters, in the present work, the effects of substituents on the , IR, ^1H and ^{13}C NMR spectra and crystal structures of a number of N-(aryl)-sucinamic acids of the general formula, i-$XC_6H_4NHCOCH_2CH_2CO_2H$ (X= H, Cl or CH_3; i = 2, 3 or 4) and i,j-$X_2C_6H_3NHCOCH_2CH_2CO_2H$ (X = Cl or CH_3; i,j = 2,3; 2,4; 2,5; 2,6; 3,4 or 3,5) have been studied. Similarly the IR, ^1H and ^{13}C NMR spectral and single crystal structural studies of several N-(aryl)-N'-(aryl)-succinamides of the general formula, (i-XC_6H_4)$NHCOCH_2CH_2CONH$(k-YC_6H_4) (X/Y = H, Cl or CH_3; i = 2, 3 or 4) have also been made and the data analysed**.**

Esters of carboxylic acids are an important group of compounds. They are used in a wide variety of chemical reactions and are also found as natural products. Esters and lactones occur widely in nature. Virtually all commercially important fats and oils from plants and animals consist almost entirely of esters derived from glycerol known as triglycerides, in which the esterifying carboxylic acids are long

chain acids of various types. Other important products from plants include tannins. Lichens produce esters formed from aromatic acids and phenols. Microorganisms produce a complex array of compounds containing the ester linkage, ranging from simple esters to macro cyclic lactones. Synthetic polyesters are extremely important polymers and are very widely used in the textile industry. Thus the chemistry of them is of interest. The crystal structures of four N-(aryl)-methyl-succinamates, $C_6H_5NHCOCH_2CH_2CO_2CH_3$; 4-$ClC_6H_4NHCOCH_2CH_2CO_2CH_3$; 2,3-$Cl_2C_6H_3$-$NHCOCH_2CH_2CO_2CH_3$ and 3,5-$Cl_2C_6H_3NHCOCH_2CH_2CO_2CH_3$, and two N-(aryl)-ethylsuccinamates, 3,4-$(CH_3)_2C_6H_3NHCOCH_2CH_2CO_2CH_2CH_3$ and 3,5-$(CH_3)_2C_6H_3NHCOCH_2CH_2CO_2CH_2CH_3$ have also been studied.

The derivatives of N-phenyl-sucinimides have good pre-and post-emergence selective herbicidal properties and they influence plant growth[5]. N-(3,5-dichloro-phenyl)-succinimide is used as a fungicide. It produces marked nephrotoxicity in rats[5]. Thus in the present study, nine N-(substitutedphenyl)-succinimides of the general formulae, i-$XC_6H_4NC_4H_4O_2$ and i,j-$X_2C_6H_3N$-$C_4H_4O_2$ (X = Cl or CH_3; i = 2, 3 or 4 & i,j = 2,3/2,4/2,6) have also been studied.

Hydrogen bonding in the structures of the compounds studied exhibit different patterns. The graph sets are used to systematically compare the hydrogen-bond patterns observed. The trends in hydrogen-bond patterns with varying substitutions in the benzene rings attached to the amido nitrogen atoms have been described.

Experimental Methods

Small single crystals of the above compounds used in X-ray diffraction studies were grown by slow evaporations of dilute solutions of the recrystallized samples of the compounds in aqueous ethanol/methanol or mixtures of toluene and methanol or acetone and chloroform. The single-crystal studies were carried out mostly at room temperature. The collected intensity data were corrected for Lorentz polarisation and absorption. The positional parameters were determined by direct methods and least squares refinement (SHELXL-97)[2].

The details of structure solutions are as follows[2] :

Measurement device: CAD-4 Diffractometer (Enraf–Nonius, 1996)/
Xcalibur Diffractometer with Sapphire CCD detector
(Oxford Diffraction, 2009).

Data collection: **CAD**-4-PC (Nonius, 1996)/
CrysAlis CCD (Oxford Diffraction, 2009);

Cell refinement: CAD-4-PC (Nonius, 1996)/
CrysAlis RED (Oxford Diffraction, 2009);

Data reduction: REDU4 (Stoe & Cie, 1987)/
CrysAlis RED (Oxford Diffraction, 2009);

Program(s) used to solve structure: SHELXS97 (Sheldrick, 2008);

Program(s) used to refine structure: SHELXL97 (Sheldrick, 2008);

Molecular graphics: PLATON (Spek, 2009); ORTEP-3 (Farrugia, 1997);
DIAMOND (Brandenburg, 2002);

Software(s) used: SHELXL97 (Sheldrick, 2008); PLATON (Spek, 2009);
WinGX (Farrugia, 1999).

Infrared spectral measurements were made on FT-IR spectrophotometers. The resolution was set to 4 cm^{-1} and the scanning range was 400 – 4000 cm^{-1}. The spectra were measured in the solid state in the form of KBr pellets.

The NMR spectral measurements were carried out on a BRUKER Ac 300F, 400MHz FT-NMR spectrometer. Both ^{1}H and ^{13}C NMR spectra were recorded in DMSO/$CDCl_3$ with tetramethylsilane (Me_4Si) as internal standard.

The results of the synthetic, spectral and structural studies[6-9] are presented and discussed in the thesis. The thesis is broadly divided into EIGHT CHAPTERS with several sections in each chapter. List of contents of the thesis are as follows:

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6.3 Infrared Spectra of N-(Aryl), N'-(Aryl)-Succinamides.
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CHAPTER 7 EFFECT OF SUBSTITUENTS ON THE CRYSTAL STRUCTURES OF N, N'-BIS(ARYL)-SUCCINAMIDES AND N-(ARYL), N'-(ARYL)-SUCCINAMIDES

7.1 A Review of Literature on the Crystal Structures of Succinamides.
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CHAPTER 8 EFFECT OF SUBSTITUENTS ON THE CRYSTAL STRUCTURES OF N-(ARYL)-SUCCINIMIDES

8.1 A Review of Literature on the Crystal Structures of Succinimides.
8.2 Preparation and Characterization of N-(Aryl)-Succnimides.
8.3 Single-Crystal Structures of N-(Aryl)-Succinimides.
8.4 Comparisons and Conclusion.

Chapter 1 gives a general introduction to the infrared and nuclear magnetic resonance spectroscopic techniques, crystal structure studies, hydrogen bonding and sketches the objectives and scope of the present work.

Chapter 2 describes the synthetic, infrared, ^{1}H and ^{13}C NMR spectral studies on twenty two substituted N-(aryl)-succinamic acids of the general formulae i-$XC_6H_4NHCOCH_2CH_2CO_2H$ (X = H, CH_3, Cl, Br or F and i = 2 / 3 / 4), i,j–$X_2C_6H_3NHCOCH_2CH_2CO_2H$ (X = CH_3 or Cl and i,j = 2,3 / 2,4 / 2,5 / 2,6 / 3,4 / 3,5) and N-(2,4,6-trimethylphenyl)-succinamic acid. The infrared spectra of all the N-(aryl)-succinamic acids have been measured in the solid state. The variations of IR absorptions of these compounds with the substitutions in the benzene ring are discussed. The variations of N-H, C=O and C-N absorption frequencies with substitutions in the phenyl ring in N-(aryl)-succinamic acids are shown in terms of bar diagrams. The ^{1}H and ^{13}C NMR spectra of the above compounds in solution have also been measured and analysed. The chemical shifts of both the aromatic protons and carbons have been calculated by three different methods and compared with the measured values. The comparison of the observed chemical shifts and the calculated values for both ^{1}H and ^{13}C NMR spectra revealed that the three procedures of calculations lead to almost the same values in most cases, indicating that the principle of additivity of the substituent effects is reasonably valid in these compounds. The variation of chemical shifts of N-H protons and C=O carbons in the side chain with substitutions in the benzene ring are shown in terms of bar diagrams.

Chapter 3 describes the crystal structures of monosubstituted N-(aryl)-succinamic acids of the general formula, i-$XC_6H_4NHCOCH_2CH_2CO_2H$ (X = H, CH_3 or Cl; i = 2, 3 or 4). The data have been analyzed to explore the effect of the site and nature of substitutions on the crystal structures of N-(aryl)-succinamic acids.

The comparison of the crystal systems of all the monosubstituted N-(aryl)-succinamic acids showed that the introduction of a chloro or methyl substituent at the meta position of NPSA changes the crystal system from monoclinic to orthorhombic, while the introduction of Cl at ortho or para position of NPSA does

not change the crystal system, but the introduction of methyl group at these positions changes the crystal system from monoclinic to triclinic crystal system. Although there are variations in the bond lengths, bond and torsion angles with the site and nature of substitutions in the phenyl ring, there are no particular trends in their variations, may be because the compounds crystallize in different crystal systems with different space groups.

The conformational changes with varying substitutions have also been considered. The conformation of amide N-H bond is anti to the C=O bond in the amide segments of all the N-(monosubstitutedphenyl)-succinamic acids. The conformation of the amide C=O and the carboxyl C=O bonds are also anti to each other in the parent, ortho and para substituted succinamic acids, while they are syn to each other in the meta-methyl and meta-chloro substituted succinamic acids. But the conformation of both the amide C=O and carboxyl C=O bonds are anti to the C-H bonds of the adjacent methylene groups, while the conformation of the C=O and O-H bonds of the acid group are syn to each other in all the monosubstituted N-(aryl)-succinamic acids. The conformation of the N-H bond is positioned syn to the ortho and meta-chloro atoms and ortho-methyl groups in the phenyl ring, while it is anti with respect to meta-methyl group.

The overall comparison of the crystallographic data and geometries of the structures including the H-bond geometry revealed that the site and nature of the substitutions in the phenyl ring of N-(monosubstitutedphenyl)-succinamic acids have significant effects on these parameters, although there are no particular trends in their variations.

Chapter 4 describes the crystal structures of disubstituted N-(aryl)-succinamic acids of the general formula, i,j-$X_2C_6H_3NHCOCH_2CH_2CO_2H$ (X= CH_3/Cl; i,j = 2,3/2,5/2,6/3,4/3,5) and N-(2,4,6-trimethylphenyl)-succinamic acid. The data have been analyzed to explore the effect of disubstitutions and trisubstitution on the crystal structures of N-(aryl)-succinamic acids.

The comparison of the crystallographic data, the packing diagrams and the hydrogen bond patterns and the other data of the N-(mono/di/trisubstitutedphenyl)-succinamic acids indicate that the the site, the nature and the quantum of

substitutions affect the structural parameters, although with the limited data it is very difficult to generalise the effects. For example, introduction of methyl group at the ortho or para position of the parent compound, N-(phenyl)-succinamic acid, changes the crystal system from monoclinic to triclinic, while the introduction of Cl at these positions has no effect on the crystal system. But the introduction of either the Cl or the methyl group at the meta position changes the crystal system from monoclinic to orthorhombic. Further, the dichloro substitutions at different positions of the parent succinamic acid do not change the crystal system. It remains to be monoclinic. But the di- and tri-methyl substitutions have different effects. The torsion angle which reflects the orientation of the side chain with respect to the phenyl ring is affected by the substitution to different extents. A closer look at the hydrogen bond patterns or the packing diagrams reveals the effect of the site, nature and the quantum of substitutions on these aspects, although it is difficult to generalise the trends with the substitutions. Generally the meta substitutions play important role as is evident from the rare anti conformation observed between the carboxyl C=O and the O-H bonds in meta disubstituted compounds, in contrast to the more common syn conformation observed between the carboxyl C=O and the O-H bonds in all other succinamic acids. Generally N-H...O H-bond lengths remain more or less the same with all the succinamic acids, while the O-H...O H-bond lengths are generally longer with the chlorosubstitutions in the phenyl ring, indicating the weakening of the O-H...O H-bond with these substitutions.

Chapter 5 describes the crystal structures of six N-(aryl)-alkylsuccinamates and analyzes the data to explore the effect of substitutions on the crystal structures of these succinamates. The structures have also been compared with the structures of the corresponding succinamic acids.

The comparison of the crystallographic data and the geometries of the structures indicates that the introduction of an alkyl group into the carboxyl group to produce esters have varying effects on the crystal data and the geometries. The significant changes are outlined as follows: The replacement of H-atom of the carboxyl group in the side chain of N-(phenyl)-succinamic acid to produce N-(phenyl)-methylsuccinamate (NPMS), changed the conformation between the amide C=O and the carboxyl C=O from anti to syn in NPMS. But in the case of N-

(4-chlorophenyl)-succinamic acid, the replacement of the H-atom in the carboxyl group by $–CH_3$ to produce N-(4-chlorophenyl)-methylsuccinamate has no effect on the conformation. Thus the introduction of Cl at para position negated the effect of methyl substitution into the carboxyl group. Further, the replacement of the H-atom in the carboxyl group by $–CH_3$ to produce N-(3,5-dichlorophenyl)-methyl-succinamate (N35DCPMSA) changes the conformation in the resulting ester group. The C=O and -O–CH_3 groups of the ester segment in N35DCPMSA are in syn position to each other, in contrast to the rare anti conformation between the C=O and O-H bonds in the corresponding succinamic acid. Esterification of the acid group also changes the H-bond geometry and the graph set notations as well. Further, the four succinamic acids crystallize in the same monoclinic crystal system, while the corresponding methyl esters crystallize in different crystal systems.

Chapter 6 describes the synthetic, infrared, ^1H and ^{13}C NMR spectral studies on twenty seven substituted N-(aryl),N'**-(**aryl**)-**succinamides of the general formula i-$XC_6H_4NHCOCH_2CH_2CONH$(j-YC_6H_4) (X/Y = H, CH_3 or Cl and i/j = 2,3 or 4).

The infrared spectra of N-(aryl)-N'-(aryl)-succinamides have been measured in the solid state. The variations of N-H, C=O and C-N IR absorptions with the substitution in the benzene rings have been considered and are shown in terms of bar diagrams. The comparison of values and the bar diagrams indicate that there are no particular trends in the variation of these frequencies on substitution of either the electron withdrawing or electron donating groups.

The ^1H and ^{13}C NMR spectra of all the compounds have been measured in solution. Further, ^1H and ^{13}C Chemical shifts of all the aromatic protons and carbons in N-(aryl), N'-(aryl)-succinamides have been calculated by adding the substituent contributions to the ^1H or ^{13}C chemical shifts of N-(phenyl), N'-(phenyl/ substitutedphenyl)-succinamide. The comparison of the calculated and the experimental chemical shifts of protons and carbons in these compounds revealed that there is a good agreement between the two sets of values.

Chapter 7 describes the crystal structures of five N,N'-bis(aryl)-succinamides of the general formula, (i-$XC_6H_4NHCOCH_2$)$_2$, and six N-(aryl), N'-

(aryl)-succinamides of the formula,i-$XC_6H_4NHCOCH_2CH_2CONH$(j-YC_6H_4). The structural data have been analyzed in comparison with those of N, N'-bis(phenyl)-succinamide to explore the effect of the nature and site of substitutions on the structural parameters of these compounds.

The comparison of the crystal structure data reveals that all the N,N'-bis(aryl)-succinamides and N-(aryl),N'-(aryl)-succinamides, except one, crystallize in monoclinic crystal systems. The behaviour of the structures can be generalised into the following three categories:

1. N-(i-X-phenyl),N'-(i-X-phenyl)-succinamides (X = H, CH_3or Cl& i = 2, 3 or 4),
2. N-(i-X-phenyl),N'-(i-Y-phenyl)-succinamides (X =CH_3, Y = Cl& i = 2, 3 or 4) and 3. N-(i-X-phenyl),N'-(j-Y-phenyl)-succinamides (i-X = H & j-Y = 4-CH_3; i-X = 2-CH_3&Y = 3-Cl andi-X = 3-CH_3& Y = 4-Cl).

In the first category of compounds,the asymmetric units of the structures contain one half-molecule with a center of symmetry at the mid-point of the central C-C bond. The N-H, C = O and C-H bonds in the C-NH-C(O)-C fragments are all anti to the adjacent bonds. However, the conformation of the N-H bonds in the amide fragments are anti to the ortho or meta-methyl groups and meta-Cl atoms in the adjacent benzene rings, but syn to the ortho-Cl atoms in the benzene rings. The dihedral angles between the benzene rings and the NH-C(O)-CH_2 fragments in the structures of NN'BSA, NN'B2MPSA, NN'B3MPSADH, NN'B4MPSA, NN'B2CPSA and NN'B3CPSA are 33.4(1), 62.1(2), 5.6(4),30.5(6), 47.0 (2) and 32.8(1)^0, respectively. The corresponding C2-C1-N1-C7 torsion angles in the structures are 37.2(1), 64.0(4), 5.1(1), 32.8(2), 47.6(6) and 35.0(3)^0. The striking deviation of angles with NN'B3MPSADH is due to the fact that it is a dihydrate unlike the other compounds which are non-hydrates.

In all the crystals, except NN'B3MPSADH, series of N-H…O intermolecular hydrogen bonds link the molecules into chains. In NN'B3MPSADH, the packing of molecules is through O—H…O and N—H…O hydrogen bonding interactions.

The behaviour of N2MPN'2CPSA, N3MPN'3CPSA and N4MPN'4CPSA, the second category of compounds, are similar in that all the three molecules contain one half-molecule in their asymmetric units with a center of symmetry at themid-point of the central C-C bond. Further, the N-H, C = O and C-H bonds in

the C-NH-C(O)-C fragments are all anti to the adjacent bonds. But the methyl and Cl groups are disorderedwith respect to the ortho / meta / para-positions of the benzene ring, withsite-occupation factors of 0.5 each.The dihedral angles between the benzene rings and the NH-C(O)-CH_2 fragments of N2MPN'2CPSA, N3MPN'3CPSA and N4MPN'4CPSA are 62.0(2), 43.5(1) and 39.9(1)^0, respectively, compared to the values of 33.4(1), 62.1(2), 5.6(4), 30.5(6), 47.0 (2) and 32.8(1)^0 for NN'BSA, NN'B2MPSA, NN'B3MPSADH, NN'B4MPSA, NN'B2CPSA and NN'B3CPSA, respectively. In the crystals, the intermolecular N-H…O hydrogen bonds link the molecules into chains.

The third category of compounds, NPN'4MPSAH, N2MPN'3CPSAH and N3MPN'4CPSAH are all monohydrates. They all show interesting behaviour. In these crystals, the water molecule accepts two and makes two hydrogen bonds. In NPN'4MPSAH, the dihedral angles between the benzene and methylbenzene groups with the mean planes of the attached NH-C(O)-CH2 fragments are 12.6 (4) ^{0} and 23.3 (3) ^{0}, respectively, while that between the two aromatic rings is 73.7 (2)^0. The dihedral angles between the chlorobenzene and methylbenzene groups with the mean planes of the attached NH–C(O)–CH2 fragments are, respectively, 9.4 (4) and 62.9 (2) ^{0} in N2MPN'3CPSAH, and 16.6 (2) and 22.8 (2) ^{0}in N3MPN'4CPSAH. In the crystals, O—H…O and N—H…O hydrogen bonds link the components into a two-dimensional networks.

It is evident from the above that the site and nature of substitutions significantly influence the structural parameters of this class of compounds. But to generalise the effects, additional data are required. Efforts are on in this direction.

Chapter 8 describes the preparation, characterization and crystal structures of nine N-(aryl)-succinimides of general formulae, i-$XC_6H_4NC_4H_4O_2$ (i = 2 / 3 / 4 and X = CH_3 / Cl) and i,j-$X_2C_6H_3NC_4H_4O_2$ (X = CH_3 and i,j = 2,3 / 2,4 / 2,6) along with a review of literature on the crystal structures of succinimides.

The crystal structures of N-(2-methylphenyl**)**-succinimide, N-(3-methyl-phenyl**)**-succinimide, N-(4-methylphenyl**)**-succinimide, N-(2-chlorophenyl**)**-succinimide, N-(3-chlorophenyl**)**-succinimide, N-(4-chlorophenyl**)**-succinimide, N-(2,3-dimethylphenyl**)**-succinimide, N-(2,4-dimethylphenyl**)**-succinimide and

N-(2,6-dimethylphenyl**)**-succinimide have been determined and the data analyzed to explore the effect of substituents on the structural parameters.

The comparison of the cell parameters of the parent and N-(mono-/di-methylphenyl)-succinimides reveals that the mono-methyl and 2,3-dimethyl-substitution in the phenyl ring has no effect on the type of crystal system. It remains to be monoclinic with $P2_1/c$ space group with the parent, N-(4-methylphenyl)- and N-(2,3-dimethylphenyl)-succinimides and $P2_1/n$ space group with N-(2-methylphenyl)- and N-(3-methylphenyl)-succinimides. In contrast to this, the halosubstitution have different effects on the crystal systems. The ortho-fluoro substitution changes the crystal system from monoclinic to orthorhombic, while the meta- and para-fluoro substitutions have no effect. The mono-chloro substitution changes the crystal system from monoclinic to the orthorhombic, irrespective of the site of mono-substitution, while mono-bromo substitutions have different effects.

It is expected that the nature, site and quantum of substitutions in the phenyl ring affect the orientation of it with respect to the succinimide ring. The orientations of these rings are measured in terms of the torsion or dihedral angles between the least-squares planes of the benzene and succinimide rings. Generally, the ortho-substitution increases the torsion angle, while the meta-substitution lowers it and the para-substitution has little effect on the torsion angle. Although there are slight variations in the bond lengths, bond angles and other torsion angles with the site and nature of substitutions in the phenyl ring, there are no particular trends in their variations. Further, it is evident from the comparison of the packing diagrams and the hydrogen bond patterns of the structures that the site and nature of substitutions in the phenyl ring change these aspects considerably, thus may be markedly affecting their activities of these biologically active compounds.

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   Acta Cryst., **E65** (2009) o388.

2. Structure Report on N-(2-Chlorophenyl)-succinamic acid,
   B. T. Gowda, S. Foro, **B. S. Saraswathi**, H. Terao & H. Fuess,
   Acta Cryst., **E65** (2009) o399.

3. Structure Report on N-(2,6-Dimethylphenyl)-succinamic acid,
   B. T. Gowda, S. Foro, **B. S. Saraswathi,** H. Terao & H. Fuess,
   Acta Cryst., **E65** (2009) o466.

4. Structure Report on 2,5-Dichloroanilinium chloride monohydrate,
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Attested by the Guide

(Mrs. Saraswathi, B. S.)

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